

Air Products and Chemicals, Inc.

Cheaper, More Efficient Oxygen Through Novel Production Method

The ability to separate oxygen from air has proven invaluable to many industries, because using pure oxygen in high-temperature furnaces improves their efficiency and reduces emissions. Although the benefits of using oxygen are substantial, its widespread use has been hindered by the high cost of oxygen-separation technologies. In 1992, Air Products and Chemicals, Inc. (APCI), a leading supplier of industrial gases, demonstrated a new approach to oxygen separation in the laboratory. APCI's approach was based on ion-transport membranes (ITMs) that efficiently produce high-purity (greater than 95 percent) oxygen at a high temperature. The company's new approach was limited to basic laboratory trials and had yet to be incorporated into a functional prototype. Therefore, in order to advance the research into this promising technology, APCI submitted a proposal to the Advanced Technology Program (ATP). In their proposal, they highlighted the potentially significant, industry-wide impacts of their proposed technology, as well as the technical risks.

In 1993, ATP awarded APCI cost-shared funding for a three-year project to advance the development of the ITM technology. Although APCI was not able to develop a functional ITM prototype by project end, the ATP-funded project did result in two achievements: the development of ceramic-steel seals and the development of designs and parameters to determine the most efficient method to remove contaminants from oxygen. These accomplishments helped to further APCI's research into oxygen-separation techniques by several years and encouraged the company's management and other agencies to support additional research and development for the ITM technology. The Department of Energy has supported the effort with a funding commitment for three separate three-year research phases between 1998 and 2007 to investigate new oxygen-separation methods.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 93-01-0041 were collected during October - December 2001.

Significant Market Exists for Lower Cost, High-Purity Oxygen

In 1991, the daily production of gaseous and liquid oxygen in the United States was nearly 450 billion cubic feet, or about 51,500 tons. Costly high-purity (greater than 95 percent) oxygen was used in many industries such as steel, glass, and aluminum production and municipal waste incineration. These industries relied on oxygen for its ability to produce high temperatures essential to the efficient operation of furnaces and incinerators. The electronics industry used ultra-high-purity oxygen to produce a thin oxide layer on the

surface of silicon wafers during the fabrication of complementary metal-oxide semiconductors and other circuits. Power-generation and oxygen-enriched combustion applications used pure oxygen in the integrated gasification combined cycle. It was predicted that the total market would grow significantly if lower cost high-purity oxygen could be produced.

Other industries that would benefit from less costly pure oxygen included waste water treatment, pulp and paper manufacturing, lake and river revitalization, and hazardous waste incineration. While these sectors' use of oxygen was minor compared to the steel industry,

they represented potential high-yield growth in an area that would positively impact the environment. However, these new industries would only benefit from the use of oxygen if new, low-cost oxygen-separation processes could be developed.

Existing Oxygen-Separation Processes Hinder Widespread Adoption

The technology used for the commercial production of oxygen from air varied. For example, cryogenic distillation was the process used in large-tonnage production (100 to 2,000 tons per day) of high-purity oxygen. For small-tonnage production (less than 100 tons per day), the pressure swing adsorption (PSA) or vacuum swing adsorption (VSA) methods were used. Processes to produce less than one ton per day were also available. However, all of these methods had drawbacks that prevented cost-effective high-purity oxygen production, and they were prohibitively expensive for many potential applications.

Since 1989, Air Products and Chemicals, Inc. (APCI), a leading supplier of industrial gases, had been pursuing unique ways to separate oxygen from air. They believed that a low-cost oxygen-separation process for scale-up purposes could be developed. APCI's proposed approach was to incorporate an ion-transport membrane (ITM) into a prototype oxygen-separation unit. They believed that only the ITM technology could achieve totally selective separation and thus a pure oxygen product. Their proposed separation process involved compressing the feed air, pulling a vacuum on the product oxygen, or a combination of the two. While this process was similar to the other separation processes used at the time, APCI calculated that the use of mixed oxide ITM could result in a cost savings of at least 30 percent over PSA, VSA, or cryogenic distillation. That significant price reduction would allow numerous additional applications for pure oxygen, and the oxygen-production industry would be revolutionized.

Funding Support Needed for High-Risk Oxygen Separation

In 1992, APCI realized that if their research into ITM technology were to continue at a sustainable pace, they would need additional capital. Although APCI's management believed in the technology, they could not

justify risking such a high percentage of their research and development (R&D) budget on a technology that was still 10 years from commercialization. In addition, the technical risks of this project were very high. Each step of the project would require the development and incorporation of many novel subcomponents into a finished product, each carrying its own obstacles and complications. Therefore, the company submitted a proposal to ATP in 1993 and was awarded a three-year project with approximately \$2 million in funding assistance.

APCI Identifies a Four-Phase Development Program

In 1989, when APCI began its R&D into the ITM technology, they envisioned a four-phase development program that would include the following:

Phase I: Fundamental Membrane/Process Development-internal funds

Phase II: Laboratory Prototype Development Unit (PDU)-with ATP funding help

Phase III: Technology Demonstration Plant-with Department of Energy (DOE) funding

Phase IV: Commercialization-internal funds

Phase I was solely funded by APCI and concluded in 1993. During that four-year phase, the company made significant technology advances in powder preparation of membrane compositions, dense-membrane fabrication, and thin-film-membrane deposition. Furthermore, APCI gained an increased understanding of the transport mechanisms of oxygen through the membrane. This, in turn, led to the development of proprietary membrane structures and ITM module designs.

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APCI viewed phase II as a crucial step in the further development of the ITM technology. This stage included the design, construction, and testing of a laboratory

PDU. To help achieve its goal for this phase, APCI contracted with Ceramatec, Inc. to aid in the development of the ITM module, which was the key component of the proposed prototype. Ceramatec, a private company based in Salt Lake City, Utah, brought extensive ceramic expertise to the project.

As the ATP project progressed during phase II, APCI and Ceramatec collectively worked on the components that would be used in the PDU. Both companies faced problems in developing several components that were integral to the success of the project. Although APCI was not able to develop a functional PDU based on the ITM technology during the ATP project, the company did develop ceramic-steel seals for the PDU and the designs and parameters to determine the most efficient method to remove contaminants from oxygen.

Moreover, additional benefits from this ATP project included confirmation of the concept of a thin-cell membrane's approach to the small-scale development of proprietary technology (which is still being pursued) and an increase in the body of knowledge and experience in the technology, which is considered invaluable by APCI. The completion of phase II in 1997 marked the end of the ATP project.

Phase III was to have begun after the ATP project was completed and was intended to benchmark the work accomplished during phase II. The construction and operation of a commercial-scale membrane unit in the range of 1 to 10 tons of oxygen per day was to have been demonstrated.

Phase IV would have launched the full commercialization of the technology. Although APCI did not develop a functional PDU, their accomplishments during this project encouraged additional R&D support that continues today. APCI continues to strive for a 30-percent cost reduction for oxygen separation. Their current strategy is to begin commercialization initiatives after this reduction in cost is achieved.

Given APCI's progress during phases I and II, the company adjusted its original timeline. Rather than move forward with a technology demonstration plant in phase III and commercialization in phase IV, the company attracted DOE funding for additional research

into creating an oxygen-separation system that could achieve a 30-percent cost reduction.

Additional Post-Project Support Advances ITM Technology

The collaboration between APCI and Ceramatec on the ITM technology continued after the ATP project, with APCI employees working on-site at Ceramatec. Furthermore, many additional participants are engaged with APCI to advance the technology and the knowledge gained during the ATP project, including Texaco, McDermott Technology, Eltron Research, Concept RERC, University of Pennsylvania, and Pennsylvania State University. Additional governmental agencies are also supporting further development of this technology.

APCI continues to leverage its ATP-funded work in its current project with DOE. The DOE project, which was awarded in 1998, is divided into three phases, each lasting three years. The first phase focused on selecting the final materials for the scale-up of a full-size ITM membrane. For phase I, the total investment by both APCI and DOE was \$25 million, with each contributing approximately \$12.5 million. The second phase of the project entails scaling up ITM modules, which comprise many small ITM membranes; demonstrating that the new modules can enable a 30-percent cost savings over conventional air-separation technologies; and illustrating that the production of five tons of high-purity oxygen a day is possible. The third phase will demonstrate that 25 tons of oxygen can be processed per day and that the new process can be used with gas turbines, an idea initially conceived during the ATP project.

Conclusion

Air Products and Chemicals, Inc. (APCI) began its research and development (R&D) of the ion-transport membranes (ITM) technology in 1989. By 1993, the company had invested approximately \$8.3 million in the development of the technology. The development of a functional laboratory prototype development unit that used the ITM technology, which was the ultimate objective of the ATP-funded project, was not successful. However, the company did achieve two

milestones during the project: the development of ceramic-steel seals and the development of designs and parameters to determine the most efficient method to remove contaminants from oxygen. These accomplishments have been instrumental in continuing the R&D for ITM technology. These seals and design and parameter methods, as well as the additional knowledge gained during the project, have enabled further R&D by APCI and others. To help disseminate this knowledge, the company published several research disclosures in two professional journals in March 1995.

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In 1998, APCI entered into a contract with the Department of Energy to continue its R&D of this technology, and the company has partnered with several businesses to further its efforts. APCI is still committed to its original objective to develop an oxygen-separation technology that will generate high-purity (greater than 95 percent) oxygen with a 30-percent cost savings over traditional processes such as pressure swing adsorption, vacuum swing adsorption, and cryogenic distillation.

PROJECT HIGHLIGHTS

Air Products and Chemicals, Inc.

Project Title: Cheaper, More Efficient Oxygen Through Novel Production Method (Energy-Efficient Oxygen Production Using Novel Ion-Transport Membranes)

Project: To design, build, and test a laboratory prototype air-separation unit for producing high-purity oxygen using high-temperature ion-transport membranes (ITMs) that results in a 30-percent savings in cost over traditional methods.

Duration: 03/01/1994-02/27/1997

ATP Number: 93-01-0041

Funding (in thousands):

ATP Final Cost	\$1,998	42%
Participant Final Cost	<u>2,721</u>	58%
Total	\$4,719	

Accomplishments: Air Products and Chemicals, Inc. (APCI) was not able to develop a prototype development unit (PDU) based on the ITM technology. However, the company developed ceramic-steel seals that will be incorporated into the PDU and developed processes to remove contaminants from oxygen. Moreover, APCI published research disclosures in March 1995 in two articles.

Commercialization Status: APCI is continuing its research and development (R&D) so that future commercialization is possible. However, the company does not intend to pursue commercialization initiatives until a 30-percent decrease in production cost is achieved.

Outlook: APCI, its subcontractor Ceramatec, its many additional business partners, and the Department of Energy (DOE) are continuing R&D of this technology. APCI has concluded the first phase of a three-phase project with DOE that began in 1998 and is expected to end in 2007.

Composite Performance Score: *

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